Standard Operating Procedure

Routine Operation of NO_2/PAN GC

For CCOS

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Revisions and Authority

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0.5	Complete sections and finalize final copy	10/27/00

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Manager

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QA Coordinator

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1. SCOPE AND APPLICATION

This SOP describes procedures for routine operation of CE-CERT NO2/PAN GC to be operated during the Central California Ozone Study (CCOS), a 3-month monitoring program.

2. SUMMARY OF METHOD

A room temperature (25 ± ^{o}C) chromatographic column is operated on a carrier stream of zero air at a flow rate of about 40 ml/min. The column effluent passes through a Luminol detector. Ambient air is drawn through a sampling loop connected to a six-port GC injection valve. The ambient sample is injected at 1-minute intervals (i.e. the sample switched briefly into the carrier stream). PAN elutes from the column several seconds after NO_2. Excess Luminol in the detector reacts quantitatively with NO_2 and with PAN, generating light that is detected by a photo multiplier tube. The intensity of the light is proportional to the amount of NO2 or PAN reactant present in the detector. The output of the PMT is amplified and fed to the NO2/PAN GC computer data acquisition and analysis system, which stores and analyzes one complete chromatogram every minute. The NO2 and PAN peaks in the chromatogram are processed to generate peak areas, a calibration factor is applied, and the resulting ambient NO2 and PAN concentrations in ppb are stored on the NO2/PAN GC computer along with the original chromatogram. The NO2 and PAN concentrations are also sent to the monitoring station data logger, along with sample mode information.
The instrument includes an automatic calibration system that includes three modes of operation: ambient sample, zero air, and span gas. This system is set to provide 10 minutes of zero air, and 10 minutes of NO2 span gas every two hours. These zero and span responses may be used to detect and compensate for calibration drift.

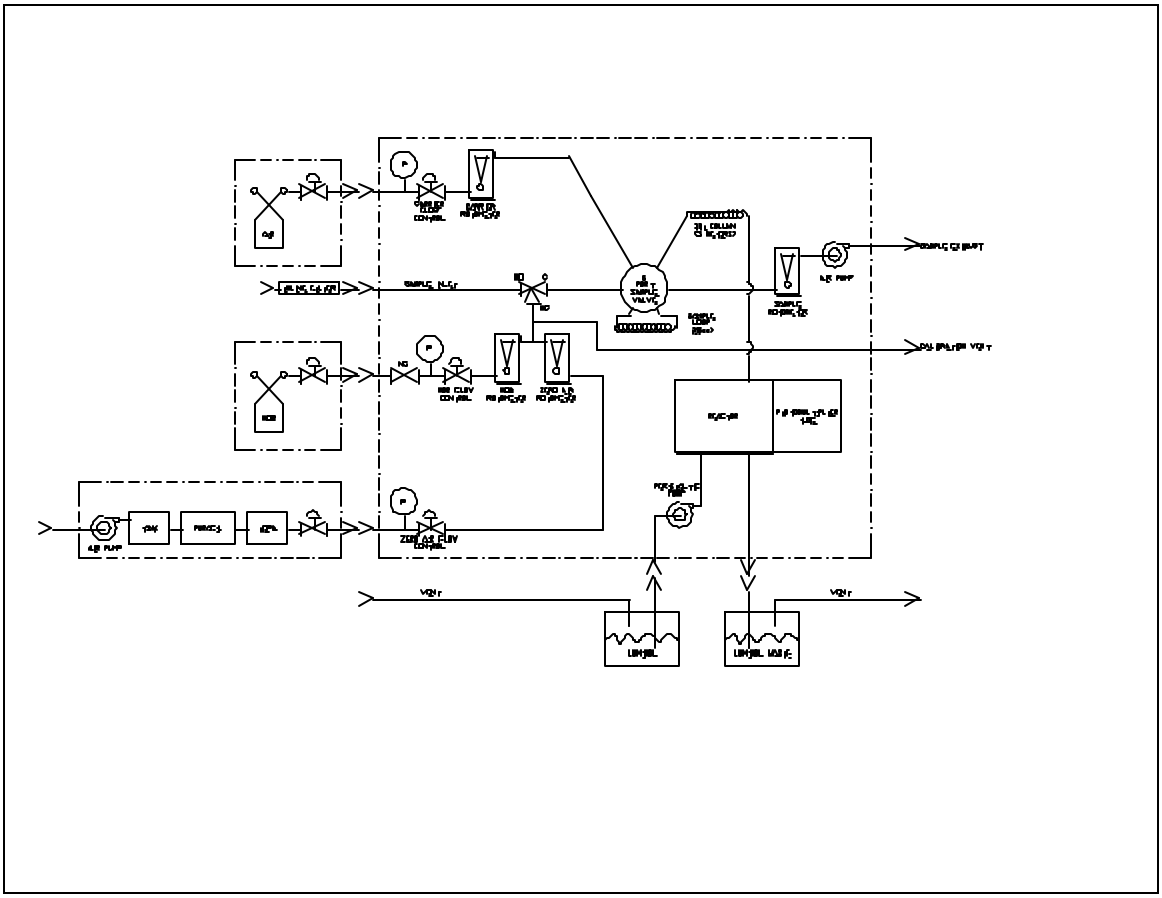

Figure 1 Schematic of NO2/PAN GC

3. DEFINITIONS

DI = deionized
GC = gas chromatograph
PAN = peroxyacetylnitrate
PMT = photo multiplier tube

4. PERSONNEL HEALTH AND SAFETY

The instrument contains line power electrical hazards. The case must be open with power applied to verify operation of the peristaltic pump. Use caution when working with the analyzer cover open.

Calibrations and routine QC require use of compressed gas cylinders. Cylinders should be properly secured and capped when not in use. Regulators should be installed and removed, following standard safety procedures. Regulators should be leak checked after installation.

5. CAUTIONS

The start up and shutdown procedures must be followed with care to prevent instrument damage. These procedures are designed to safely prevent crystallization of salt within the system. Following the procedures incorrectly or in the wrong order could result in salt crystallization. Correction of salt crystallization is an extensive task requiring disassembly and cleaning of the system.

The vents from the waste collection and the reagent source bottles connected to the system should be scrubbed to avoid contamination of the site with Luminol vapors

The liquid waste and excess reagents must not be disposed of on site. The waste should be returned to the field operations manager for disposal.

During the automated calibration cycle, the excess flow from the calibration system is about 10 lpm and includes of NO2 at a concentration of about 80 ppb. Care should be taken to ensure that the excess flow from the calibration system does not affect sample inlets of any other NOx or NOy measurements on site.

6. INTERFERENCES

Not specified.

7. PERSONNEL QUALIFICATIONS

Equipment operators should be experienced ambient air quality technicians. The technicians should receive instruction on routine operations by the CE-CERT staff performing the initial installation and setup.

8. EQUIPMENT, SUPPLIES, REAGENTS

8.1 Working Reagents and Supplies

- Luminol solution
- Carrier air: zero air cylinder with single-stage regulator
- Auto calibration gas: gas cylinder, NO_2 in N_2, with single-stage SS regulator.

8.2 Maintenance Supplies

- DI water. Water from a laboratory grade deionization system is adequate.
- NO2/PAN GC repair kit:
    1) 3 ml syringe
    2) silicon tubing (6”)
    3) 1/8 Teflon tubing (2’)
    4) Allen wrench for detector 9/64
    5) Wrenches 5/16x1/4, 7/16x3/8, 1/2x9/16, 19/32x11/16, and 5/8x3/4
    6) Crescent wrench 3”
    7) Fuses one 10A, one 800mA, both 250V, slow-blow (GMC or GDC)
    8) Short column material
    9) Column adapter: GC column to 1/8” tubing
    10) Ferrule for column adapter
    11) 1/16, 1/8, and 1/4 nuts and ferrules
    12) filters: two Millipore LS 47mm, 5µ pore size

13) 0-50 flow meter (Dwyer RMA-151 or equivalent)
14) 1/8 & 3/16 Tygon tubing
15) Teflon tape
16) Cable ties
17) Replacement peristaltic pump tubing
18) Razor blades-2 ea

8.3 Quarterly QC Equipment and Supplies

- Dilution calibrator
- Certified cylinder of NO_2 in N_2
- PAN source (uncalibrated). PAN may be generated by injecting liquid PAN and dilution air from the Calibration Zero Air system into a 100-literTeflon bag. PAN may also be generated by injecting liquid acetaldehyde and dilution air into a 100-liter Teflon bag, then exposing the bag to direct sunlight for 30 minutes.
- TECO 42 CY NOy analyzer (use the in-station analyzer)

9. SITE AND EQUIPMENT PREPARATION

The system must be installed in a temperature-controlled shelter.

The sample inlet should be connected to the station manifold so that it receives daily, automated checks from the in-station auto calibration system

The system must be positioned so that the waste line drains to a waste collection bottle below the level of the analyzer. No part of this waste line may rise above the level of the analyzer waste output connection.

The system must be position so that the Luminol reagent supply bottle is approximately level with the analyzer reagent input connection. If the Luminol supply bottle is below the level of the analyzer, the peristaltic pump may not be able to maintain flow.

10. CALIBRATION AND QC CHECKS

10.1 Frequencies/Overview

Baseline Checks
The sampling interval of the analyzer is one chromatogram every minute. Each chromatogram includes a determination of baseline. Drift or noise in the baseline indicates a problem with Luminol or carrier airflow rate. These QC data are collected and stored every minute, but are not collected by the site data logger. Thus they are available for review as frequently as site visits are performed.

2-hourly checks
The auto calibration cycle is a self test which includes one zero check and one span check every two hours. These QC data are collected by the station data logger. They are available for review as frequently as the data logger is polled (approximately daily).

Once Daily Checks
Data from the field site are polled daily by the field operations managers. QC checks performed daily include:

review response to the 2-hourly zero/span
review diurnal patterns in NO_2 and PAN
review response to the daily zero (dry pure zero air) and span check (where available)

Where possible, the analyzer sample inlet should be drawn from a station manifold that is connected to an automated station calibration system. The station calibration system should supply zero air and NO2 span gas once daily. The analyzer data must be polled and checked daily, even if the station provides no automated daily zero and span.

Weekly Checks
Check gas cylinders: NO2, Carrier air
Check Calibration Zero Air system scrubber check analyzer settings
Refill Luminol reagent
Evaluate chromatogram shape and baseline

Monthly checks
Change inlet filter

Quarterly
perform independent multipoint calibration with NO2 and PAN
perform independent airflow rate checks

Instructions for performing the QC and the maintenance procedures are given in Section 10.2. Tolerances for the QC procedures are shown in Table 1.

10.2 QC Procedures

10.2.1 Daily Checks

1. Poll data logger and review QC data as well as diurnal patterns daily (weekdays)

Table 1 QC Tolerances

QC Check	Tolerance
2-hour zero air, scrubbed ambient	0 ppb ± 2ppb
2-hour span NO2	Set point (~50 ppb) ± 5 ppb
Daily dry zero	0 ± 2 ppb
Daily NO2 span	Set point (~450 ppb) +/- 5%
Carrier Air Cylinder Pressure	> 500 psi
Carrier Air Pressure	25 psi ± 1 psi
NO2 Span Cylinder Pressure	> 1500 psi
NO2 Span Pressure	15 psi ± 1 psi
Carrier flow	Set point (~30 ccm) ± 3 ccm
Zero air flow	8 lpm ± 0.3 lpm
Span flow	40 ± 5.0 ccm

10.2.2 Weekly Checks

The checks described here should be performed on a weekly basis. At minimum, these checks must be performed every 10 days or else the analyzer will run out of reagent, which could result in damage to the instrument requiring extensive repairs.

1. Fill out the identification data indicated on the Weekly Checklist form
   site,
   date,
   time, (circle time zone: PDT or PST)
   project,
   technician,
   instrument ID
   NO2 cal gas cylinder certification data

2. Fill out the following gas supply data on the Weekly Checklist Form
   NO2 cylinder pressure
   NO2 cylinder regulated output pressure
   Carrier Air cylinder pressure
   Carrier Air cylinder regulated output pressure

3. Record all software settings on page 2 of the Weekly checklist form. The software settings are shown in the Setup window. Figure 2 shows an example of the Setup window. To display the Setup window, click the button labeled SETUP in the Main window.

4. Observe the instrument in normal Sampling mode.

   Figure 3 shows an example of a good chromatogram. The chromatogram should show an NO2 peak, then a PAN peak, and then a flat baseline. Between and after the peaks, the chromatograph baseline should be flat. The raw signal level of the baseline should be less than 1000 counts per 100 ms. The raw signal level can be read against the left-hand axis of the graph, and is also displayed in the box

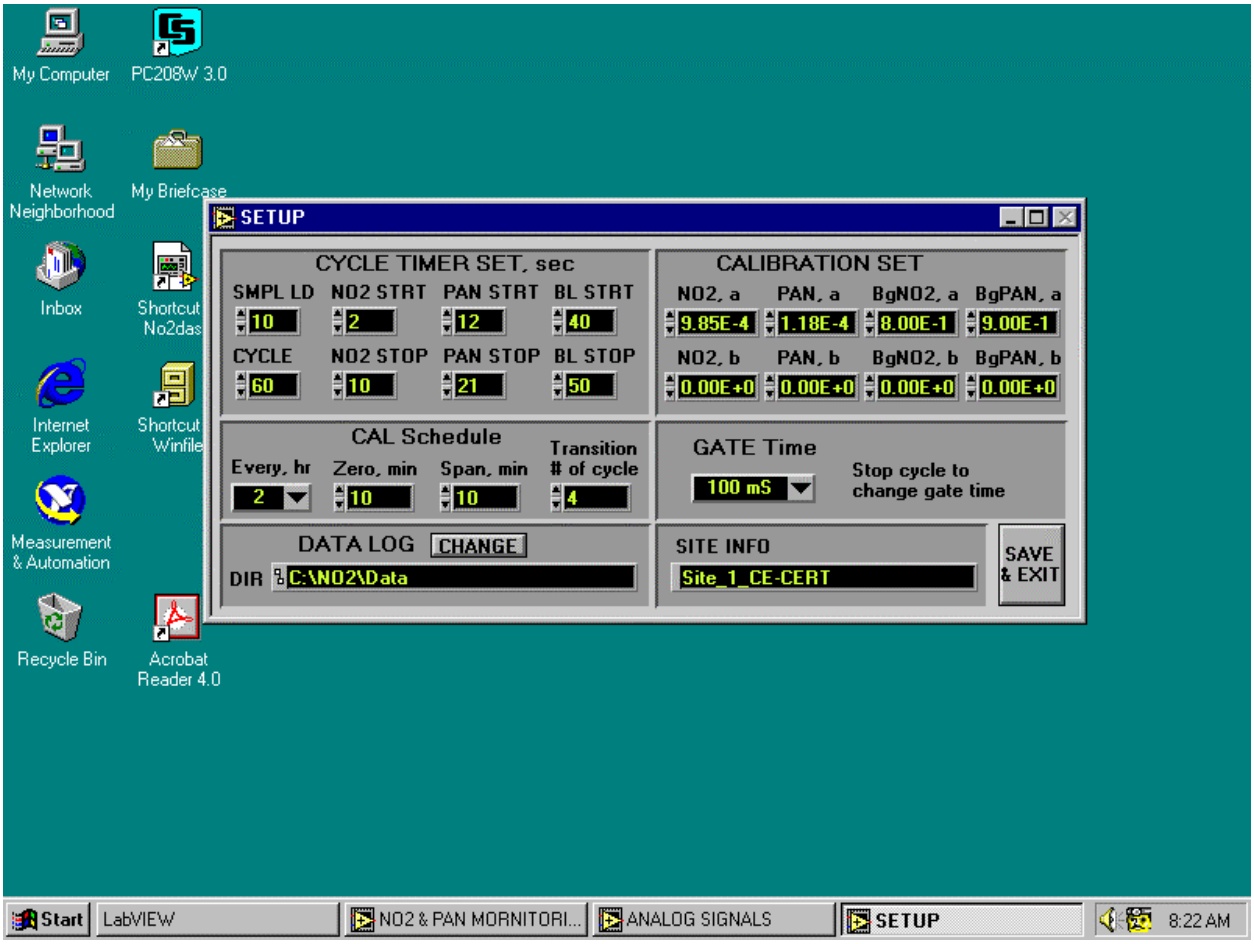

Figure 2 Setup Window

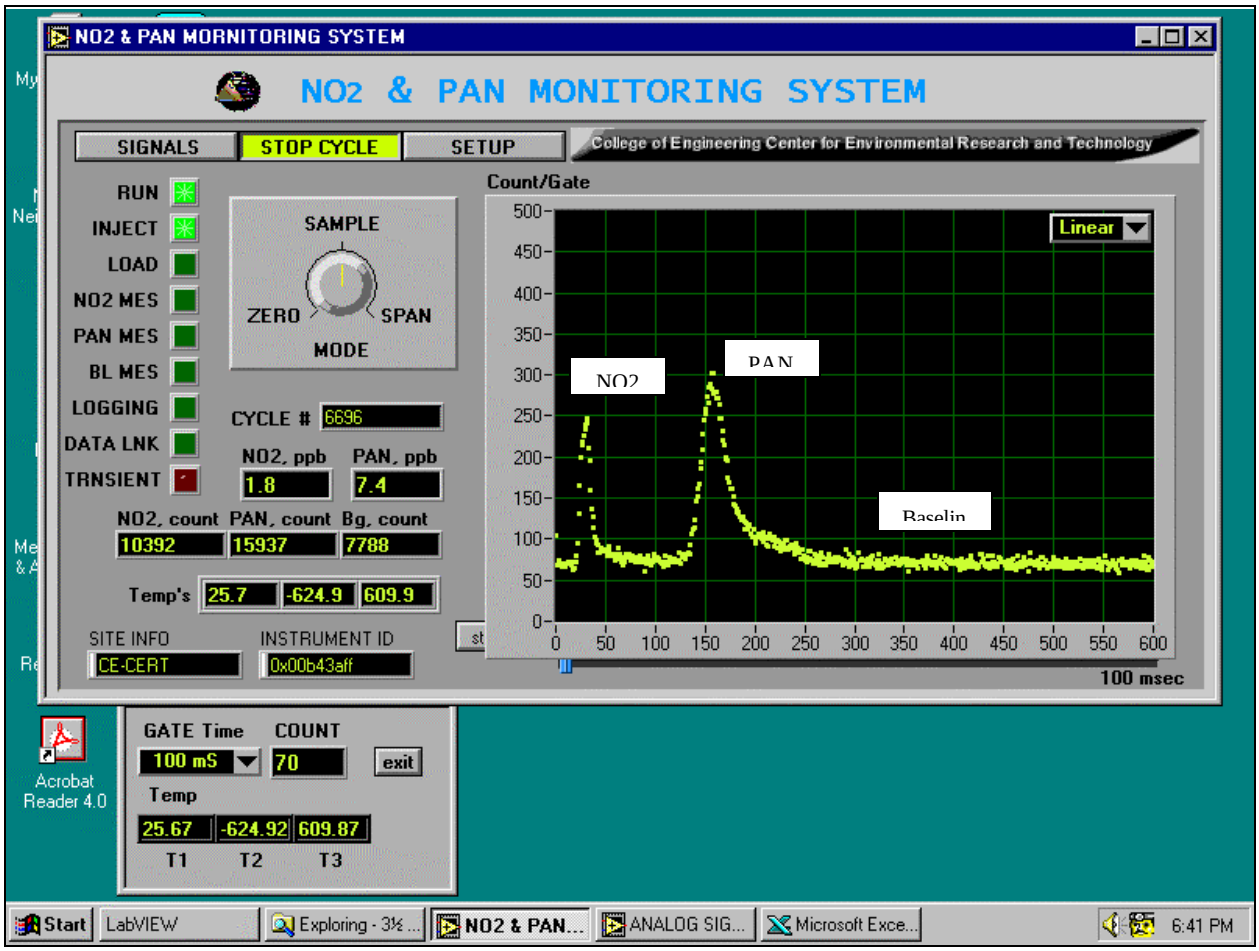

Figure 3 Analyzer Main window showing example of a good chromatogram.

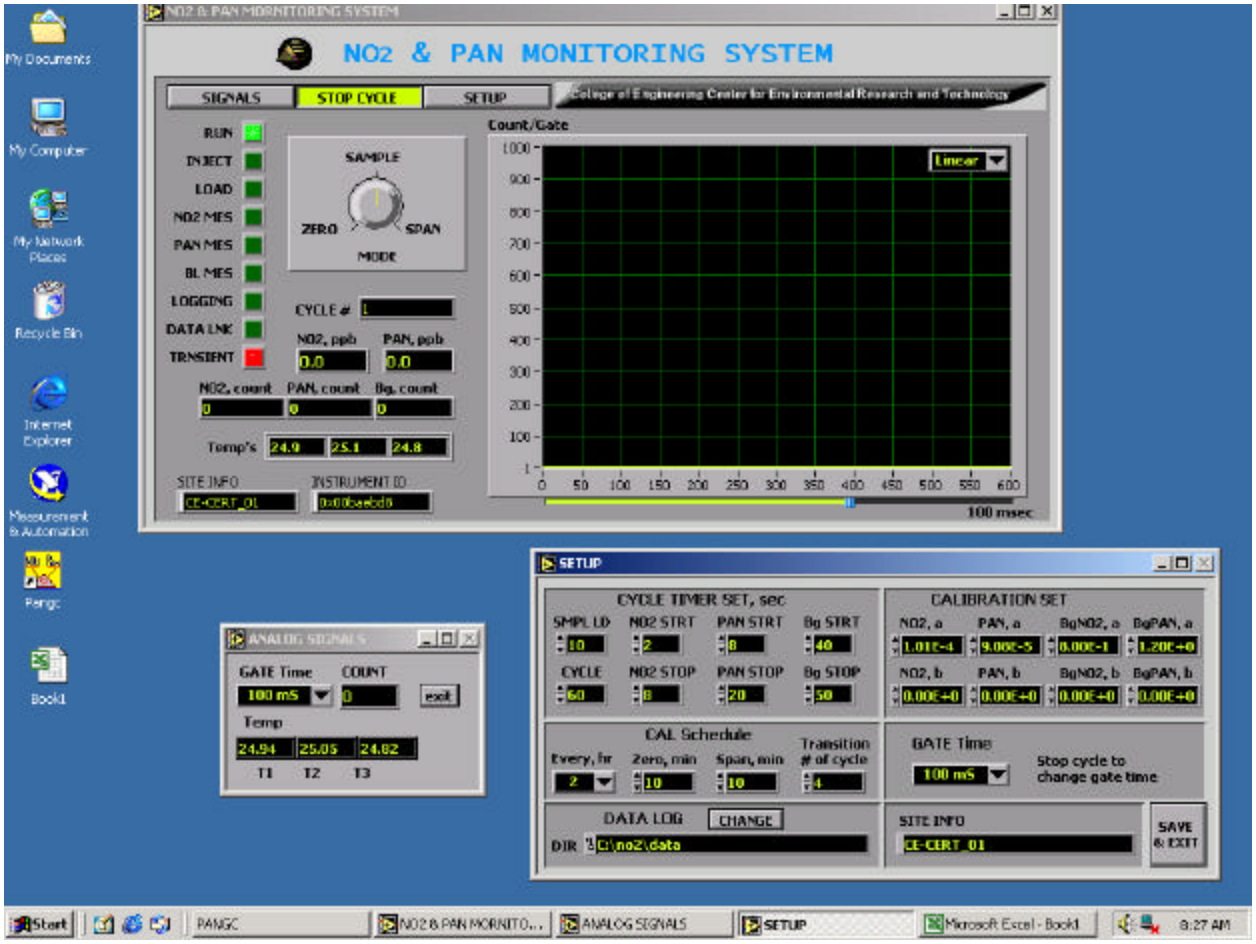

Figure 4 Analyzer Main window. Place holder for example of a bad baseline

labeled "COUNT" in the Analog Signals window. The corresponding integrated background should be less than 100,000. The integrated background is displayed in the box labeled "Bg, count" in the Main system window. (The main system window is labeled "NO2 & PAN MONITORING SYSTEM".) Deviations from the expected shape include completely flat response (no peaks), an elevated sinusoidal baseline, or a noisy erratic baseline. See Section 15 to troubleshoot these situations.

Fill out the following information. (Note: The carrier gas is Air. The front panel may be incorrectly labeled with "N_2" or "N_2 Carrier ".) The analyzer responses are read from the main software window and are recorded in the section labeled Analyzer Response Table on the Weekly Checklist form.

Sample flow rate
Carrier Gas front panel pressure
Carrier Gas flow rate
Chromatograph shape
Chromatograph baseline shape
Chromatograph baseline raw level

Analyzer response in SAMPLE mode:
- NO2, ppb
- PAN, ppb
- NO2, count
- PAN, count
- Bg, count

If the flows or pressures are not within tolerance, then record the initial values on the weekly checklist sheet, adjust to the correct values, record the new values on the weekly checklist sheet, and note your adjustment in the comment section.

5. Observe a 2-hourly zero/span cycle. The analyzer will switch to zero mode 10 minutes prior to the even hours. It will switch to span mode on the even hours, and will switch to sample mode at 10 minutes after the even hour. The current sampling mode is displayed in the main software window. Record the gas pressures, gas flow rates, and the system responses during zero mode and span mode on the Weekly Checklist Form. The gas pressures and flow rates may be recorded as soon as the analyzer switches into the appropriate mode. However, the analyzer response for first 5 minutes of each sampling mode should be ignored. Therefore, wait 5 minutes before recording the analyzer responses from the Main software window. The data to be recorded are:

   Zero Air front panel pressure
   Zero Air front panel flow rate
   Analyzer response in ZERO mode:
   - NO2, ppb
   - PAN, ppb
   - NO2, count
   - PAN, count
   - Bg, count

   Cal Gas front panel pressure
   Cal Gas front panel flow rate
   Analyzer response in SPAN mode:
   - NO2, ppb
   - PAN, ppb
   - NO2, count
   - PAN, count
   - Bg, count

6. Calculate the ratio of analyzer span response to the target span concentration. Record the ratio on the Weekly Checklist form. The NO2 ppb response observed during the automatic 2-hourly span check should agree with the target span value to with 5%. The target span value for each analyzer is recorded on the Analyzer Settings Sheet that accompanied the analyzer during setup.

7. Perform the Maintenance checks described in Section 11.2 and record the results on the Weekly Checklist form. These checks include the following:
   - Refill Luminol
   - Replace Purafil if 100% brown

Replace Teflon filter if more than 30 days old
Replace Calibration system filters if more than 90 days old

8. Perform Final Review of Weekly Checklist

Compare instrument responses with the tolerances shown in Table 1. The table shows nominal set point values for several items. During installation and setup, an Analyzer Settings Sheet is provided with each analyzer. That sheet specifies the correct settings for that individual analyzer. Report out-of-tolerance readings immediately to staff at CE-CERT by telephone. Contact at least one of the following personnel: Dennis Fitz, 909-781-5781; David Pankratz, 909-781-5754; John Collins, 909-781-5793.

Verify that no entries on the Weekly checklist are blank.

Sign the weekly checklist with your initials.

10.2.3 Monthly Checks

Drain liquid from the zero air system tank once a month.

10.2.4 Quarterly Checks

QC checks by external group

- Auto calibrator flow rates
- Multipoint NO2 calibration
- Multipoint PAN verification

11. INSTRUMENT OPERATION

General Procedures for Non-toxic Compressed Gas cylinders.

1. Strap the cylinders securely to appropriate support before removing the cylinder cap.
2. Remove the cylinder cap.
3. Crack the cylinder valve very briefly to blow any dirt from CGA fitting.
4. Connect the regulator tightly to the cylinder valve. (Use a wrench).
5. Back the regulator pressure control all the way out (counter clockwise).
6. Close the regulator outlet valve.
7. Open the cylinder valve briefly then close it (Observe that pressure increases on the cylinder pressure gage).
8. Turn the regulator pressure control clockwise until outlet pressure reads 10 psi.
9. Open the regulator outlet valve to bleed off the gas.
10. Close the regulator outlet valve.
11. Go back to step 5 and repeat the regulator purge two more times.
12. Back the regulator pressure control all the way out.
13. Make sure the regulator outlet vale is closed.
14. Open the cylinder valve fully open, then back off one-half turn.

15. Adjust the regulator pressure control until the output pressure is at the desired set point.
16. Use Snoop to check all connections for leaks. Tighten any loose connections.

11.1 Startup

During initial startup, begin with all power to analyzer disconnected. Perform the start up connections and checks in the following order.

Inspection

1. Remove the cover from the PC case.
2. Check that the PC boards are seated securely and there are no loose connections.
3. Remove the cover from the analyzer.
4. Check that all fittings are snug.
5. Leave the covers off for now.

Gas Connections

1. Place the 47 mm inlet filter at the ambient sampling point, upstream of the sampling line.
2. The sample line should have a residence time of less than 10 seconds from the ambient sampling point to the back of the analyzer. For a sample flow rate of 100 cm^3/min the residence times for common tubing sizes are a follows:

OD	ID	Length	Time
1/4“	3/16”	1.0’	3.26 sec
1/4”	2/16”	1.0’	1.45 sec
1/8”	1/16”	1.0’	0.36 sec
1/4“	3/16”	3.1’	10.0 sec
1/4”	2/16”	6.9’	10.0 sec
1/8”	1/16”	27.6’	10.0 sec

3. Connect the sampling line from the outlet of the 47-mm sample inlet filter to the tubing extending from the drilled though port labeled Sample Inlet on the back of the analyzer.
4. Vent the port on the back of the analyzer labeled Cal Gas Outlet to the station exhaust manifold or to a location outside the shelter far form the station inlet line.
5. Vent the port on the back of the analyzer labeled Sample Exhaust to the station exhaust manifold or to a location outside the shelter far form the station inlet line.
6. Connect the port labeled Zero Air Inlet on the back of the analyzer to the outlet of the Calibration Zero Air system.
7. Connect the SS NO2 regulator to the NO2 cal gas cylinder using proper procedures for gas cylinders.
8. Connect the outlet of the NO2 cal gas regulator to the port labeled Cal Gas Inlet on the back of the analyzer using PFA Teflon line.
9. Connect the Air regulator to the Carrier Air gas cylinder using proper procedures for gas cylinders.
10. Connect the outlet of the Air regulator to the port labeled N2 on the back of the analyzer using Teflon line. (This port is incorrectly labeled N2. It should be labeled Carrier Gas Inlet).

Adjustments and Leak Checks

1. Open the NO2 cal gas regulator outlet valve.
2. Adjust the NO2 cal gas regulator control until the regulator outlet valve reads 15 psi. This regulator will need further adjustment once the analyzer is put into span mode.
3. Open the Carrier Air gas regulator outlet valve.
4. Adjust the cylinder regulator control until the carrier gas pressure displayed on the front panel of the analyzer reads 25 psi.
5. Adjust the front panel carrier gas control until the carrier gas is flowing at the rate indicated on that analyzer settings sheet that accompanied the analyzer (nominally 40 ccm).
6. Make sure all power is disconnected from unit.
7. Use snoop to check all connections. Tighten as needed.

Liquid Connections

1. Connect the port labeled Exhaust Reservoir to the Waste bottle. This waste line must never rise above the level of the Exhaust Reservoir outlet port on the back of the analyzer. Do NOT submerge the outlet of the waste line in the collected waste.
2. Connect the port labeled Luminol Inlet to the Luminol supply reservoir. The bottom of the Luminol supply bottle MUST be positioned 6 to 18 inches above the bottom of the analyzer. (If the bottle is to low, the pump may lose prime. If the bottle is too high, excess flow may flood the reaction chamber causing a fluctuating elevated baseline.
3. Verify that the peristaltic pump controls are set as follows
    a. Direction: Forward
    b. Speed Class: Slow
    c. Speed Setting: 0.0
4. Pull up the plastic cover from the peristaltic pump and lift the tubing off of the peristaltic cam
5. Disconnect the 1/16" Luminol inlet Teflon line from the 1/16" Swagelok union at the inlet side of the reactor (the top connection to the reactor
6. Connect the 5-ml syringe to this line and draw Luminol through this line from the supply reservoir, until it reaches the syringe.
7. Disconnect the syringe and reconnect the supply line to the inlet of the reactor
8. Replace the tubing over the peristaltic cam

Electrical connections

1. Make sure the analyzer switch is in the 0 (OFF) position.
2. Connect the blue power cable from back of the analyzer to the Cal Zero Air system pump.
3. Connect the AC power cords to the analyzer, the computer, and the monitor.
4. Connect the 68-pin cable from the analyzer to the computer.
5. Connect the RS-232 cable from the serial port on the back of the analyzer to the Campbel data logger. (The analog terminal strip on the back of the analyzer is not used.)
6. Connect the mouse and keyboard to the computer. The mouse connection is green and is on top. The keyboard connection is below the mouse connection and is purple.

Power On

1. Turn on the main power to the analyzer. (Switch on back of analyzer to "1" (ON)).
2. Adjust the sample flow rate to 100 ccm
3. Verify that the peristaltic pump is rotating.
4. Turn on the computer by momentarily depressing the power on switch.

5. Verify that the computer boots up into Windows 2000.
6. Verify that the program no2gc begins running after about 1 to 3 minutes.
7. If the computer displa ys an error message:
    a. Continue to click OK or cancel until error messages are cleared.
    b. Close the program, clicking OK until all program windows are closed.
    c. Start the program no2gc by double clicking its icon on the desktop
8. Verify that the computer clock shows the correct date and time. Correct if needed by double-clicking on the clock display in the lower right hand corner of the screen.

Software Settings

1. Click on "Setup" to display the Setup window.
2. Verify that the values displayed in each Setup value match those shown on the Analyzer Settings Sheet accompanying the analyzer.
3. If any settings need to be adjusted, click on "Stop Cycle" to halt the collection of chromatograms. (The only setting that should need to be changed is "Site Name.")
4. Change the settings as needed.
5. Once all settings are correct, press the Enter key. (Pressing "Save and Exit" will NOT correctly enter the new settings).
6. Once the Enter key has been pressed, click on Save and Exit. (This will insure that the new settings will be loaded the next time that the software program is started.)
7. CLICK ON "Start Cycle." This will cause the software to begin collecting chromatograms at 60-second intervals.

Initial Verification

1. About 30 minutes after powering on the analyzer, briefly remove the outlet of the waste line from the waste reservoir and check that bubbles are appearing at the end of the waste line. The presence of bubbles indicates that Luminol reagent and carrier air are flowing through the reactor. If no bubbles are being generated check the carrier gas flow and the Luminol flow.
2. check being generated. verify that Luminol is flowing through the system by Remove the outlet of the waste line Verify that the

Calibration Adjustments

After the system is running, it will automatically go through a zero/span cycle every two hours. You must wait for a zero cycle to make the following adjustments.

1. Wait for the system to enter Zero mode.
2. Adjust the front panel Zero Air regulator until the front panel regulator reads 15 psi.
3. Adjust the front panel zero flow rate controller until the rotameter indicates 8.0 lpm.
4. Wait for the system to enter Span mode.
5. Adjust the Span cylinder regulator until the front panel regulator reads 15 psi.
6. Adjust the front panel Span flow rate control until the span rotameter reads a 40 ccm.

11.2 Routine Operation

The instrument operates unattended, but must be serviced at weekly intervals. The operations that must be performed on a weekly basis include:

- Check/refill Luminol supply. Rotate the bottle to remove the Luminol bottle cap to avoid crimping the Teflon line. To avoid loosing the pump prime, do not lift the Teflon line out of the Luminol.
- Check/refill Calibration Zero Air system scrubber (Purafil)

- Perform Weekly QC checks
- Report results of checks to field operations manager and to CE-CERT
- Transmit copies of data files to field operations manager and to CE-CERT

Luminol Supply refill
The old Luminol solution may be left in the supply bottle. Fill the supply bottle with fresh Luminol up to the mark (approximately to the shoulder of the bottle). Check off this action on the weekly checklist form.

Purafil scrubber refill
When all of the Purafil has turned brown, replace it with fresh Purafil: Unplug the Calibration Zero Air System pump from its power source. Remove the cartridge from the system. Open the cartridge, discard the used purafil to regular trash, and replace with fresh purafil. Pack the cartridge full so that channeling does not occur...Close the cartridge, replace it in the Calibration Zero Air system , and reconnect power to the system. (Purafil turns from purplish red to brown as it is consumed, but remains effective for removing NO2 even when it has turned brown. Thus the change does not need to be made before all of the Purafil turns brown).

11.3 Shutdown

Perform a final calibration (quarterly check) before proceeding with shutdown procedures.

1. Ensure that the following components are labeled with the monitoring site ID before proceeding with shutdown:
    a. Main analyzer unit
    b. Computer
    c. NO2 span gas cylinder
    d. Calibration zero air system
2. Close NO2 Span gas cylinder valve
3. Remove the Luminol supply line from the Luminol supply reservoir, and quickly place this line into a reservoir of DI water. (Avoid leaving the line inlet open to atmosphere. Leaving the line open to atmosphere will draw air into the system, and may cause the pump to lose prime.)
4. Wait approximately 3 hours until the chromatographic response goes to zero.
5. Click “Stop cycle” in the main software window.
6. Close the no2gc program.
7. Turn off the computer.
8. Remove the Luminol supply line inlet from DI water and leave open to atmosphere.
9. Turn off power to the analyzer
10. Close the Carrier Air cylinder valve.
11. Label the carrier gas line with “Carrier Air”
12. Label the NO2 Span gas line with ‘NO2 Span”
13. Disconnect and pack the following items:
    a. Sample inlet filter holder
    b. Sample line
    c. Span Gas supply line
    d. Span Gas regulator
    e. Carrier Air supply line
    f. Carrier Air regulator
    g. Calibration Zero Air supply line

h. Luminol supply bottle
i. Three (3) power cords

14. Cap the NO2 Span gas and Carrier Air cylinders
15. Pack and return the following items:
    a. the package put together in step 16
    b. the Span cylinder
    c. the main analyzer
    d. the computer
    e. the Calibration Zero Air system

In the comments section of the Quarterly QC Check data sheet, record the time that the analyzer was turned off.

12. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

No samples are stored in this procedure. Sample is drawn directly from the ambient air into the diffusion scrubber for analysis.

13. SAMPLE PREPARATION

There is no sample preparation for this method.

14. PREVENTIVE MAINTENENACE AND REPAIRS

The following operations should be performed quarterly in conjunction with the quarterly QC check, or more frequently as indicated by troubleshooting.

- Replace 47-mm Sample Inlet filter
- Replace peristaltic pump tubing
- Replace calibration zero air system filters

Sample Inlet filter replacement

The filter change should be accomplished as quickly as possible to minimize the amount of unfiltered air drawn into the sample line. Remove the filter holder from the inlet line and open it using the plastic wrenches supplied. Discard the old filter. Replace with new 47-mm Teflon filter. Us the plastic wrenches to tighten the filter holder. Replace the filter holder on the sample line, connecting the exit of the filter holder to the inlet of the sample line. Mark the date of the filter change on the weekly checklist sheet.

15. TROUBLESHOOTING

Potential anomalous responses include:

1. Flat response, i.e. no peaks above baseline:
   The most like cause for a completely flat baseline is lack of flow through the detector. Verify that both carrier air and Luminol are flowing out of the waste line.

2. High sinusoidal baseline:
   The most likely cause of high sinusoidal baseline is excess Luminol flow through the detector. Verify that the Luminol reservoir is located at the proper height relative the analyzer, that the peristaltic pump is set at the correct speed, and that carrier gas is flowing at about 30 ccm.
3. Very erratic noisy baseline:
   Intermittent flow of Luminol and air bubbles through the detector can cause a very erratic baseline. Check the Luminol supply. Look at the peristaltic pump tubing to determine if bubbles are being pumped through the system. Find and correct any air leaks.

16. DATA ACQUISITION, CALCULATIONS, AND DATA REDUCTION

The PMT signal is measured as a pulse rate proportional to the intensity of light emitted by the Luminol reaction. The pulses are counted and summed at 0.1-second intervals. The instantaneous count for the most recent 0.1-second interval is displayed in the Analog Signals window, and is plotted versus time on the graph in the Main window. During the course of each chromatogram, the counts are summed over three time intervals.. The nominal gate times defining these intervals are as follows:

- NO_2 seconds 2.0 through 8.0
- PAN seconds 8.0 through 20.0
- BKG seconds 40.0 through 50.0

The particular start and stop times for each individual analyzer may vary from these nominal times. The correct gate times for each analyzer are included on the settings sheet accompanying that analyzer. The total signal counts for each time window during the last chromatographic cycle are displayed in the Main window.

Concentrations of NO2 and PAN are determined by calculating the net signal response, and then applying a calibration equation to the net signal response. Because the three time intervals have different durations, the background count must be scaled to the correct time duration before it is subtracted from the NO2 and PAN counts. The following equations accomplish the calculation of ambient NO2 and PAN concentrations in units of ppb:

$$BKG_{NO2} = (A_{BgNO2})*(BKG_{counts}) + B_{bgNO2}$$
$$BKG_{PAN} = (A_{BgPAN})*(BKG_{counts}) + B_{bgPAN}$$

$$NO2(ppb) = (A_{NO2})*(NO2_{counts} - BKG_{NO2}) + B_{NO2}$$
$$PAN(ppb) = (A_{PAN})*(PAN_{counts} - BKG_{PAN}) + B_{PAN}$$

The *A* and *B* coefficients are unique to each analyzer and are provided on the Settings Sheet accompanying the analyzer. The coefficients are displayed in the Setup window. The NO_2 and PAN concentrations from the most recent chromatographic cycle are displayed in the Main window in units of ppb.

17. COMPUTER HARDWARE AND SOFTWARE

Hardware

- Pentium class personal computer with PCI bus
- National Instruments PCI-6023E multifunction I/O card

Software

- Windows 2000
- Microsoft Excel 97 or later version
- National Instruments drivers for I/O board
- Executable program: no2gc

18. DATA MANAGEMENT AND RECORDS MANAGEMENT

See QIWP.

19. METHOD PERFORMANCE

CE-CERT does not yet have sufficient experience operating these instruments to assess the typical accuracy and precision achieved by following the procedures described in this SOP.

20. POLLUTION PREVENTION

Not applicable.

21. WASTE MANAGEMENT

The analyzer waste stream and excess reagents should be collected in a bottle with a scrubbed vent. These wastes should not be disposed of on site. Returned to the waste to CE-CERT for proper disposal.

22. REFERENCES

Nees, Monica (USEPA) (1993). Quality Assurance Handbook for Air Pollution Measurement Systems, Vol. I: A Field Guide to Environmental Quality Assurance. EPA-600/R-94/038a, EPA, ORD, Research Triangle Park, 1993.

USEPA (1994). Quality Assurance Handbook for Air Pollution Measurement Systems, Vol. II: Ambient Air Specific Methods (Interim Edition). EPA-600/R-94/038b, ORD, Research Triangle Park, NC.

CE_CERT PAN/NO2 GC Weekly Checklist (page 1 of 2)

Date: _____ **Project:** _____
Technician: _____ **Site:** _____
Time: _____ PDT/PST **S/N:** _____

NO2 Cylinder ID

ID: _____
NO2 conc _____ ppm (~10 ppm)
NO conc: _____ ppm (<0.1 ppm)
Cert date: _____ < 6 months

Cylinder Pressures

Carrier gas cylinder pressure:	_____	(psi)	> 500
Carrier gas regulator pressure	_____	(psi)	25 +/- 1
NO2 cylinder pressure:	_____	(psi)	>500
NO2 regulator pressure	_____	(psi)	15 +/- 1

SAMPLE Mode checks

Sample Air flow rate	_____	ccm	200
Carrier gas front panel pressure	_____	(psi)	25 +/- 1
Carrier gas flow rate	_____		30
Chromatogram shape normal?	_____	yes/no	yes
Baseline flat?	_____	yes/no	yes
Baseline raw level	_____	counts/100 ms	<1000

ZERO mode pressures and flows

Zero Air front panel pressure	_____	psi	20 +/- 1
Zero air Flow	_____	lpm	8

SPAN mode pressures and flows

Cal gas (NO2) front panel pressure	_____	(psi)	15 +/- 1
NO2 Span flow	_____	ball height	40

Analyzer Responses

Mode	BKG counts	NO2 counts	PAN counts	NO2 ppb	PAN ppb
SAMPLE					
ZERO					
SPAN					

Span Calculation

NO2 response ppb/NO2 setpoint ppb _____ ratio 1 +/- 0.05

CE_CERT PAN/NO2 GC Weekly Checklist (page 2 of 2)

Maintenance information:

Teflon filter date:	_____	Date last changed	< 30 days
Zero Air Purafil Date	_____	Date last changed	< 90 days
Zero Air filter date:	_____	Date last checked	< 90 days
Luminol Level	_____	% of bottle	90% of bottle

Analyzer Sofware Settings

Cycle Timing (seconds)

start	0
Sample load	_____
Cycle	_____
NO2 start	_____
NO2 stop	_____
PAN start	_____
PAN stop	_____
BKG start	_____
BKG stop	_____
cal factors	_____
gate time (msec)	_____

Calibration Factors

A_NO2	_____
B_NO2	_____
A_PAN	_____
B_PAN	_____
A_Bg_NO2	_____
B_Bg_NO2	_____
A_Bg_PAN	_____
B_Bg_PAN	_____

Cal Schedule

Every (hr):	_____
Zero (min):	_____
Span (min):	_____
Transition cycles (#):	_____

Identification

Software version:	_____
Directory location:	_____
Site info:	_____

Comments _____